

Received April 13, 2015; reviewed; accepted October 16, 2015

SYNERGISTIC EFFECT OF A MIXTURE OF DODECYLAMINE AND KEROSENE ON SEPARATION OF MAGNETITE ORE

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Abstract: In this study, it was discovered that a mixture of dodecylamine (DDA)-kerosene demonstrated a stronger collecting ability for pure quartz and higher selectivity for magnetite ore. The relationship between the DDA-kerosene composition and mineral flotation performance on reverse flotation of magnetite ore was investigated. The results showed that floatability of the floating material increased when the DDA consumption increased. Specifically, during flotation, the fine particles (-0.074 mm fraction) selectively floated with DDA-kerosene. The difficult to float coarser particles (+0.074 mm fraction) with high content of quartz, required higher DDA dosage. However, higher DDA input during the flotation process did not necessarily yield better outcomes. The best results were obtained for 40/60 amine-to-oil ratio, as the quartz floated markedly with smaller magnetite loss and the flotation behavior was superior to pure DDA. Magnetite concentrates with 1.83% SiO₂ and 71.04% Fe were produced using 120 g/Mg of DDA-kerosene (W_{DDA}=40%).

Keywords: *DDA-kerosene, magnetite ore, quartz, flotation*

Introduction

Quartz is the most primary gangue mineral in an iron ore. Reverse flotation to remove quartz has been proved to be a very effective technique in the industry. Reverse cationic flotation of quartz uses mainly fatty amines and their derivatives as a collector, e.g. mono-amine, di-amine, and ether amine or a mixture of di-amine and mono-amine (Araujo et al., 2005). In order to reduce the reagent costs and enhance the collector effectiveness in flotation of oxidized ores, a mixture of reagents with different molecular structures is commonly used in the industry (Filippov and Houot, 1997; Rao and Forssberg, 1997; Sis and Chander, 2003; Filippov et al., 2010). It is reported that the combination of di-amine and monoamine increased quartz recovery (Papini et al., 2001). The presence of alcohol molecule decreased electrostatic

repulsion of charged amine cations in the adsorption layer and led to formation of the closely packed adsorption layer (Vidyadhar et al., 2002, 2003; Filippov et al., 2014). Fuel oil emulsified with amine can reduce collector consumption (Pereira, 2003).

It has been shown that nonpolar oils can be used either as a collector or a collector extender in flotation of many different types of minerals such as naturally hydrophobic coal (Sonmez and Cebeci, 2006), sulfur and molybdenite (Rubio et al., 2007; He et al., 2011), hydrophilic oxides zinc ores and hematite (Pereira and Peres, 2005; Pereira, 2003), hydrophilic salt type minerals phosphate ores, silicates, mica and feldspar (Fenske, 1956; Malghan, 1976; Sis and Chander, 2003). A number of different benefits have been observed as a result of the use of nonpolar oils as the flotation reagents. Firstly, according to Seitz and Kawatra (1986), co-adsorption of oil and collector on the mineral surface resulted in reduction of collector dosage requirement. The nonpolar oils are less expensive reagents and considerable cost savings can be achieved. Secondly, it increased the recovery of hydrophobic minerals due to improvement of air bubble-particle adhesion. It can result in a more highly mineralized froth, which drains more easily and it also reduces the initial entrainment of fine hydrophilic gangue by reducing the necessary retention time. Thirdly, the over-frothing phenomenon due to the presence of slimes can be either reduced or eliminated through agglomeration of slimes. Fourthly, the coarse particle recovery can be improved without the attendant increase in the recovery of undesirable particles, which typically caused by the increased collector dosage. Taggart (1945) firstly proposed fuel oil as an amine chain extender. The addition of fuel oil rendered unnecessary the pH control in calamine flotation. Araujo and Souza (1997) reported that reduction in amine consumption was achieved without affecting the metallurgical recovery when adding fuel oil in iron flotation. Fuel oil was also used in anionic flotation of iron ore emulsified with fatty acids (Glembotsky, 1963). It is reported that fuel oil added in phosphate can decrease fatty acid consumption and reduce the excess foaming of oleate collector (Sis and Chander, 2003). Pereira (2003) reported the possibility of a partial replacement of ether amine with non-polar oils during reverse cationic flotation. Emulsification of the oil phase in the amine solution was a key technology. The best results were obtained with addition of diesel oil to ether amine with an 80/20 amine-to-oil ratio using Tergitol TMN-10 as an emulsifier (Pereira, 2003).

In our previous study, modification of oil assisted flotation of quartz particles was proposed. It was based on introduction of a mixture of dodecylamine (DDA) and kerosene as the collector with DDA cationic reagent coated on kerosene to hydrophilic quartz particles in the pulp (Liu et al., 2014). It is observed that agglomeration of fine quartz minerals in the presence of miscible DDA-Kerosene collector led to the formation of very large compact agglomerates resulting in increasing hydrophobicity of particles, and it was feasible that kerosene partially substituted for DDA in reverse flotation of magnetic separation concentrate. The purpose of this paper is to

investigate the synergistic effect of DDA-kerosene to magnetite ore and the relationship between the collector composition and the mineral flotation performance.

Experimental

Materials

A pure quartz (99%) sample from Lingshou (Hebei Province, PR China) was crushed and ground to the size range of $-74\ \mu\text{m}$, and the volume median diameter (d_{50}) of the fine quartz was $27.6\ \mu\text{m}$. Pure magnetite with 71.65% Fe was obtained from TISCO Jianshan iron ore processing plant (Shanxi Province, PR China). The size fraction of $-0.038\ \text{mm}$ was up to 70%.

The real ore sample was the fifth staged low intensity magnetic separation concentrate from Jianshan iron ore processing plant of TISCO (Shanxi Province, PR China). The sample was analyzed by using a Shimadzu Labx XRD-6000 diffractometer with $\text{CuK}\alpha$ radiation and Ni for filter. Figure 1 illustrates the X-ray diffraction pattern of the sample. The XRD analysis demonstrated that the main minerals of the ore sample were magnetite (Fe_4O_3) and quartz (SiO_2). The magnetite ore sample contains 65.13% Fe and 9.33% SiO_2 .

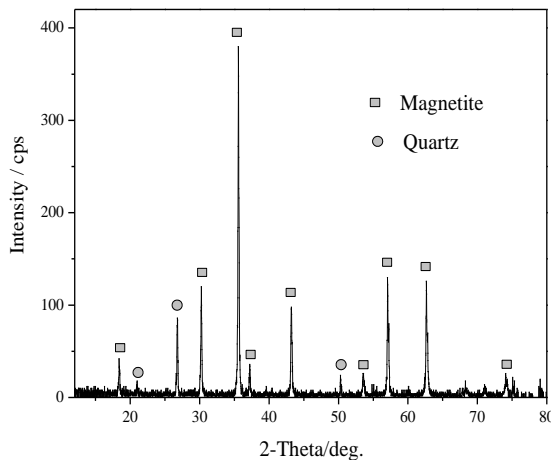


Fig. 1. XRD patterns of magnetite ore sample

The size analysis of the sample is presented in Table 1. The mineral distribution was strongly affected by the particle size. The total iron content increased and silica content decreased with decrease of the particle size. The majority of the material existed in the relative size range of $-0.038\ \text{mm}$ with a yield of 62.59%. Magnetite mainly distributed in the $-0.038\ \text{mm}$ fraction, and quartz mainly distributed in the $0.045\sim 0.074$ and $-0.038\ \text{mm}$ size fractions. The size fraction of $-0.038\ \text{mm}$ had high iron and low silica contents, since it was represented mainly by liberated grains of Fe-

oxides. The +0.045 mm size fraction comprised locked particles of magnetite, as well as aggregates of gangue, which was evidenced by the high content of SiO₂, and especially in the +0.074 mm fraction the silica content was up to 43.98%.

Table 1. Result of size analyses of magnetite ore

Fraction /mm	Weight (%)	Grade (%)		Distribution degree (%)	
		Fe	SiO ₂	Fe	SiO ₂
+0.074	2.19	39.03	43.98	1.31	10.32
0.045–0.074	18.04	58.09	18.87	16.09	36.47
0.038–0.045	17.14	66.01	9.07	17.37	16.66
–0.038	62.59	67.87	5.45	66.23	36.55
Total (calc.)	99.96	65.13	9.33	100.00	100.00

Dodecylamine (DDA) with analytical grade was obtained from Tianjin Guangfu Chemical Research Institute. The reagent grade kerosene regarded as oily solvent was supplied by Aladdin Reagent. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used as pH regulators and methyl isobutyl carbinol (MIBC) was used as a frother. HCl, NaOH and MIBC were of analytical grade obtained from Aladdin Reagent. Different concentrations of DDA were dissolved in kerosene and stirred with a magnetic stirrer at 40 °C until a homogeneous sample was obtained. The binary system collector was miscible DDA-kerosene. Our previous study showed that DDA-kerosene binary system was a partially miscible system, whose upper critical solution temperature was 34°C (Liu et al., 2014). Above the upper critical solution temperature DDA and kerosene were fully miscible in any proportion without addition of a co-solvent. Tap water used in the flotation tests, and its pH was 7.63. The hardness of water was 400 mg/dm³ (CaCO₃) and salinity was 4.5 ‰. The dominant dissolved cations were Cu²⁺ and Zn²⁺, with contents of 0.9 and 0.75mg/ dm³, respectively.

Methods

Flotation tests of pure quartz and magnetite were carried out in a XFG-1.5 flotation machine with a 1.5 dm³ cell. The stirring speed was set as 1500 rpm, with pulp density of 20%, and temperature of pulp was 25 °C. Collector conditioning time was 2 min. MIBC was added after the collector. The dosage of MIBC was 40 g/Mg, and agitation time with MIBC was 15 sec. The pH values were regulated by addition of NaOH and HCl. For magnetite ore, the pulp density was 30% and the flotation procedure was the same as for the single mineral.

Standard Taylor screens were used for the size analysis to obtain the Fe grade and SiO₂ content in different particle sizes. The flotation feed, froth and concentrate samples were wet screened at different size fractions of +74, -74 + 45, - 45 + 38 and - 38 μm. The sample was dried and dispersed by using a sample splitting device, and

then a 100 g sample was extracted for the size analysis. After drying and weighing, the screened samples were analyzed for the Fe grade and SiO₂ content.

Efficiency E was calculated using the following equation: $E = \gamma (\beta - \alpha) \beta_m / \alpha (\beta_m - \alpha) \cdot 100\%$ where α is the grade of raw ore, β iron concentrate grade, γ iron concentrate yield, β_m theoretical content of pure mineral, which for pure magnetite is 72.41%.

Results and discussions

Flotation of pure quartz and magnetite

In order to clarify the collecting ability of miscible DDA-kerosene, a comparative test of pure DDA and DDA-kerosene was carried out. The preliminary studies showed that over 80% of quartz floated with 60 g/Mg DDA. To fully understand the interaction of DDA and kerosene, the intermediate reagent dosage was selected. Different weight ratios of DDA-kerosene mixture were used in flotation at natural pH, while keeping the total collector dosage constant at 60 g/Mg. The effect of mixing collector composition on flotation is presented in Figure 2. It is seen that the collecting capability of the respective collectors follow the trend: DDA-kerosene > DDA under the same DDA dosage. It is well known that nonpolar oil does not adsorb onto natural hydrophilic minerals in the absence of collector and no flotation is achieved with nonpolar oil itself. The synergistic effect of DDA and kerosene was found compared with DDA alone.

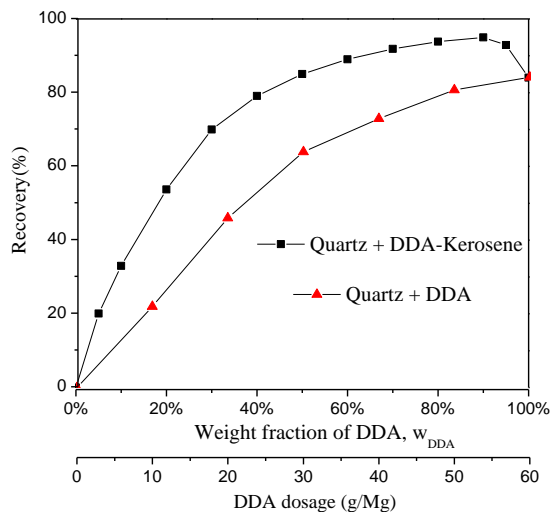


Fig. 2. Effect of DDA-kerosene composition and DDA dosage on flotation recovery of quartz

As expected, with the increase of the DDA proportion in DDA-kerosene, the recovery of quartz increased. The result showed that a small amount of kerosene can greatly improve the flotation behavior and the improvement was produced by kerosene. It should be pointed out that with the 50/50 DDA/kerosene ratio the recovery for quartz achieved approximately the same with pure DDA. There was slight improvement in the recovery by further increasing the DDA dosage. As kerosene was cheaper than DDA, DDA-kerosene utilized in the subsequent tests was a mixture in the weight ratio of 50/50.

The effect of pH on the flotation recovery of quartz and magnetite with DDA-HCl and 50% DDA-kerosene was presented in Figure 3. The collector dosage was 80 g/Mg. It was observed that the recovery-pH curve of quartz and magnetite with DDA-kerosene was consistent with DDA. This suggested that the role of DDA-kerosene was dependent on the DDA performance as expected. The most efficient flotation was obtained at neutral pH, where the recovery of quartz with DDA was higher than DDA-kerosene under alkaline pH condition. Conversely at low pH the recovery of both minerals was smaller than at neutral pH. It was found that the recovery of quartz with DDA-kerosene was nearly fairly with DDA, while more than 20% magnetite floated with DDA in the pH range of 5~8. It indicates that DDA-kerosene has an equivalent collecting capacity and markedly better selectivity compared with DDA.

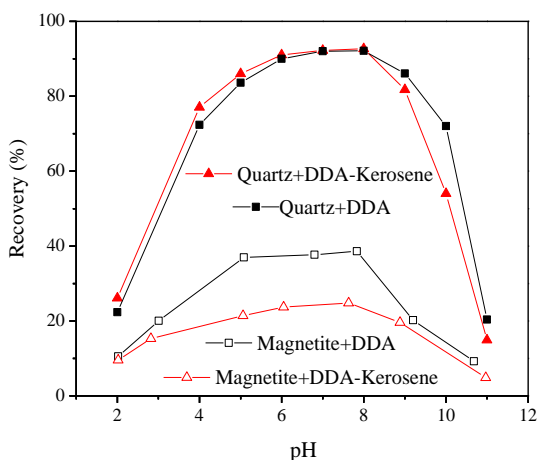


Fig. 3. Flotation results of pure quartz and magnetite with DDA and DDA-kerosene

Flotation of magnetite ore

The flotation data of pure minerals showed that DDA-kerosene performed better than DDA-HCl, as it had the equivalent collecting capability of quartz, while a relatively higher selectivity for magnetite. The effect of DDA-kerosene on flotation of magnetite ore was investigated and is shown in Table 2. The experiments demonstrated that the optimum DDA dosage was 120 g/Mg. Different weight ratios of DDA and kerosene

were used in ore flotation at natural pH, while keeping the total collector dosage constant at 120 g/Mg. As depicted in Table 2, the froth products recovery increased as the fraction of DDA increased in the DDA-kerosene. The experimental results showed that in the concentrate product, the Fe grade increased with the increase of DDA proportion, and SiO₂ content declined with the increase of DDA. The same phenomenon appeared in the froth product. It indicated that the increase of DDA addition can greatly improve the yield of froth product, however this increase was non-selective. Magnetite can float together with quartz. In general, the concentrate grade increased with the increase of the DDA content, while the recovery decreased significantly.

Table 2. Result of flotation of magnetite ore with DDA-kerosene and DDA at natural pH

Reagent	Products	Yields (%)	Grade (%)		Recovery (%)	
			Fe	SiO ₂	Fe	SiO ₂
20% DDA-kerosene	Froth	12.20	47.4	31.61	8.90	40.71
	Concentrate	87.80	67.42	6.41	91.10	59.29
	Total (calc.)	100.00	64.98	9.48	100.00	100.00
30% DDA-kerosene	Froth	24.09	48.96	29.57	18.16	76.46
	Concentrate	75.91	70.05	2.89	81.84	23.54
	Total (calc.)	100.00	64.97	9.32	100.00	100.00
40% DDA-kerosene	Froth	29.84	51.18	27.15	23.46	86.32
	Concentrate	70.16	71.04	1.83	76.54	13.68
	Total (calc.)	100.00	65.11	9.39	100.00	100.00
50% DDA-kerosene	Froth	31.32	51.92	25.92	24.97	86.72
	Concentrate	68.68	71.15	1.81	75.03	13.28
	Total (calc.)	100.00	65.13	9.36	100.00	100.00
60% DDA-kerosene	Froth	38.39	54.81	22.56	32.39	89.15
	Concentrate	61.61	71.25	1.71	67.61	10.85
	Total (calc.)	100.00	64.94	9.71	100.00	100.00
80% DDA-kerosene	Froth	43.85	56.87	19.84	38.31	92.26
	Concentrate	56.15	71.52	1.3	61.69	7.74
	Total (calc.)	100.00	65.10	9.43	100.00	100.00
90% DDA-kerosene	Froth	46.44	57.06	18.81	40.81	93.68
	Concentrate	53.56	71.75	1.11	59.19	6.32
	Total (calc.)	100.00	64.93	9.32	100.00	100.00
100% DDA	Froth	30.12	51.92	26.05	24.06	83.12
	Concentrate	69.88	70.65	2.28	75.94	16.88
	Total (calc.)	100.00	65.01	9.44	100.00	100.00

Figure 4 shows the flotation results of different composition of DDA-kerosene and corresponding pure DDA. The result indicated that the optimum proportion of DDA

was 40%. The concentrate grade increased from 68 to 71%. With further increase of DDA content, the difference between Fe grade of concentrates by DDA-kerosene and DDA was decreased gradually. Figure 5 shows that higher recoveries were obtained in the presence of kerosene with insignificant change in the Fe grade.

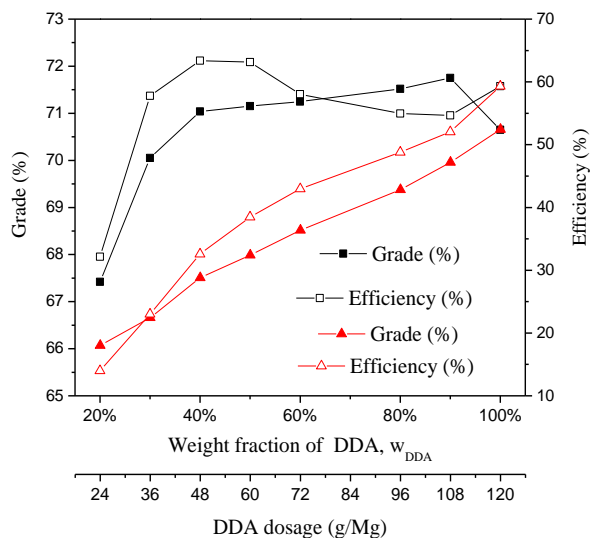


Fig. 4. Effect of DDA-kerosene composition and DDA dosage on flotation of magnetite ore

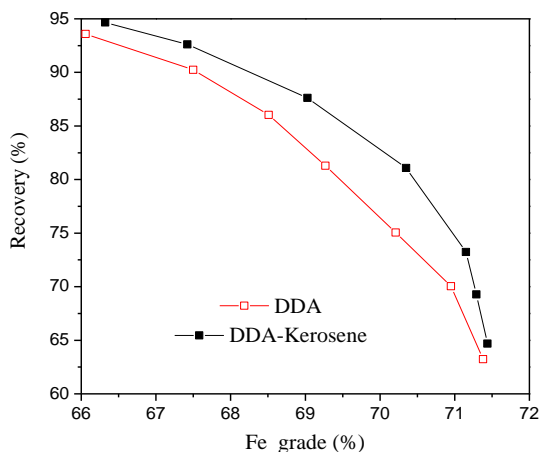


Fig. 5. Grade-recovery plot of flotation data obtained for magnetic separation concentrate

Our previous study (Liu et al., 2014) showed that the presence of DDA-kerosene collector led to formation of very large compact agglomerates of fine quartz. The

obvious synergistic effect of fine quartz was observed compared with coarse quartz particles. The characteristics of the flotation product were analyzed to demonstrate the effect of DDA-kerosene composition on the flotation recovery of magnetite and quartz in different size fraction of magnetite ore.

Figures 6 and 7 show quartz and magnetite recovery versus DDA composition ($W_{\text{DDA}} = 20\text{--}90\%$; point and line) curves of different narrow size fractions compared with pure DDA ($W_{\text{DDA}} = 100\%$; point). As shown, 20% DDA proportion was insufficient, and as a result, the quartz recovery was very low for each size fraction. Figure 6 shows that there was an abrupt increase in the quartz recovery in the DDA proportion range of 20 to 40%, with the further increase of DDA ($W_{\text{DDA}} = 40\text{--}90\%$) consumption, quartz recovery of each size fraction increased slightly, and the increased recovery of coarse fraction was greater than that of fine fraction. The recovery of -0.074 mm fractions was almost constant in the DDA proportion range of 40 to 90%. The recovery of each size fraction from higher to lower was listed as following: -0.038 , $0.045\text{--}0.074$ and $0.038\text{--}0.045$ mm indicating that the fine particle (-0.038 mm fraction) would selectively float. The $+0.074$ mm size fraction recovery increased from 77 to 94% indicating that the coarser size fraction with high SiO_2 content was difficult to float and needed higher DDA dosage. Figure 6 also demonstrates that compared with pure DDA the miscible DDA-kerosene ($W_{\text{DDA}}=40\%\sim 90\%$) was successful to float fine quartz, as the -0.074 mm size fraction recovery was higher than that with pure DDA. It demonstrated that for both pure quartz and magnetite ore, DDA-kerosene exhibited an obvious synergistic effect on the fine particles compared with the coarse particles. The data plotted as the grade-recovery relation (Fig. 8) show that higher recoveries were obtained in the presence of DDA-kerosene ($W_{\text{DDA}} = 40\sim 80\%$) compared with pure DDA.

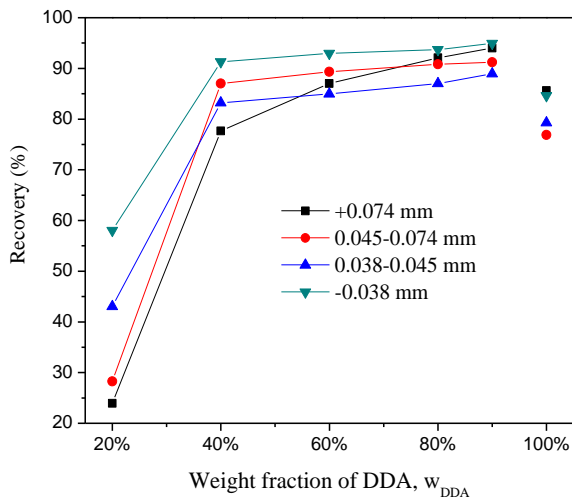


Fig. 6. SiO_2 recovery of froth product in different size fractions as function of composition of DDA-kerosene

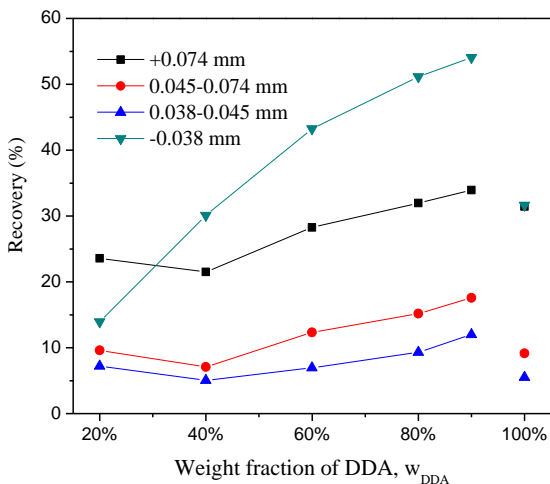


Fig. 7. Fe recovery of froth product in different size fractions as function of the composition of DDA-kerosene

Figure 7 demonstrates that the magnetite loss in each size fraction increased with the increment of DDA consumption excluding the insufficient content of 20% DDA proportion. With the increase of DDA consumption, the recovery of each fraction from higher to lower was listed as follow: -0.038, +0.074, 0.045-0.074 and 0.038-0.045 mm. The +0.074 and 0.045-0.074 mm size fractions mainly comprised locked particles of magnetite, as well as aggregates of gangue. When quartz floated, magnetite would also float, and as a result, magnetite loss of this fraction was inevitable. While the -0.038 mm fraction had the highest increased rate indicating that more finer magnetite floated with more DDA, non-selective collecting phenomenon of magnetite would happen. It means that in order to flotation of the coarse quartz particles, the DDA consumption would increase, however the finer magnetite loss would magnify with the DDA consumption rising. Compared with DDA-kerosene(60-90%), the decrease in Fe recovery with 100% DDA was obvious. The results indicated that the best results were obtained when adding kerosene to DDA with a 40/60 amine/oil ratio, as the quartz floated markedly with less magnetite loss and the flotation behavior was superior to pure DDA. The data were re-plotted to exhibit grade-recovery relation in Fig. 9 which depicts that the higher recoveries were obtained in the presence of DDA-kerosene ($W_{DDA}=60-80\%$) compared with pure DDA with the same Fe grade. The results also indicated that the best results were obtained with DDA-kerosene ($W_{DDA}=40\%$) as a result of less magnetite loss compared with pure DDA.

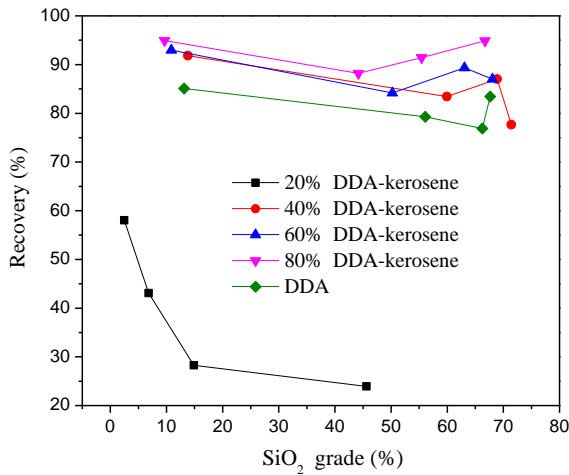


Fig. 8. SiO₂ grade-recovery plot obtained from froth product in different size fraction with DDA-kerosene. SiO₂ grade in the X-axis from low to high was: -0.038, 0.038-0.045, 0.045-0.074, +0.074 mm size fraction

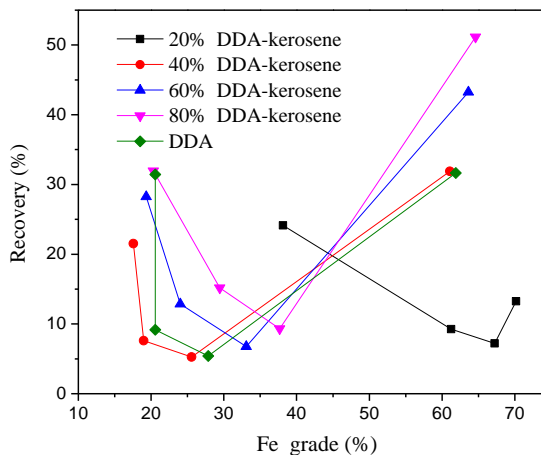


Fig. 9. Fe grade-recovery plot obtained from froth product in different size fraction with DDA-kerosene. Fe grade in the X-axis from low to high was: +0.074, 0.045-0.074, 0.038-0.045, -0.038 mm size fractions

The content of SiO₂ and Fe in the concentrate of each narrow size fraction at different DDA-kerosene composition ($W_{DDA}=20\%-90\%$; point and line) compared with pure DDA ($W_{DDA}=100\%$; point) is shown in Figs 10 and 11, respectively. The finer the particles, the higher the Fe grade was. The SiO₂ content in each size fraction decreased with the increase of DDA consumption. Figure 10 demonstrates that with 40% DDA-kerosene the SiO₂ content of the -0.045 mm size fraction decreased to less

than 2%. With higher DDA consumption, the SiO_2 content of the coarse size fraction declined, especially the +0.074 mm fraction. The content of SiO_2 and Fe versus DDA composition curves presented in Figs. 10 and 11 also indicate that the recovery of coarser particles (mainly +0.074 mm fraction) needs more DDA consumption. Figures 12 and 13, which depict DDA-kerosene ($W_{\text{DDA}}=40\%-90\%$), shows better performances in the floatation of magnetite and less content of quartz in the concentrate.

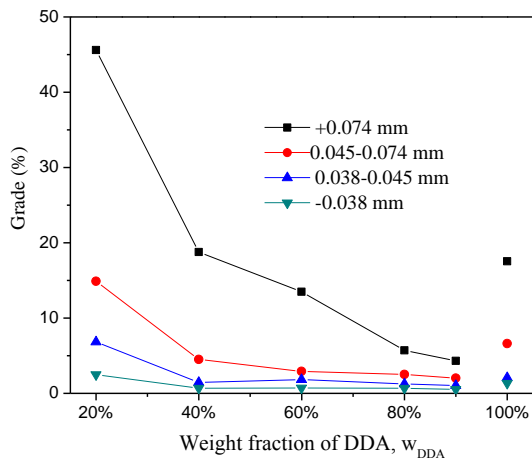


Fig. 10. SiO_2 grade in different size fractions as function of composition of DDA-kerosene

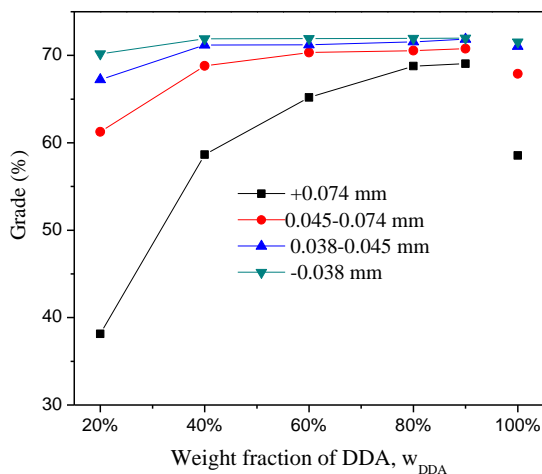


Fig. 11. Fe grade in different size fractions as function of composition of DDA-kerosene

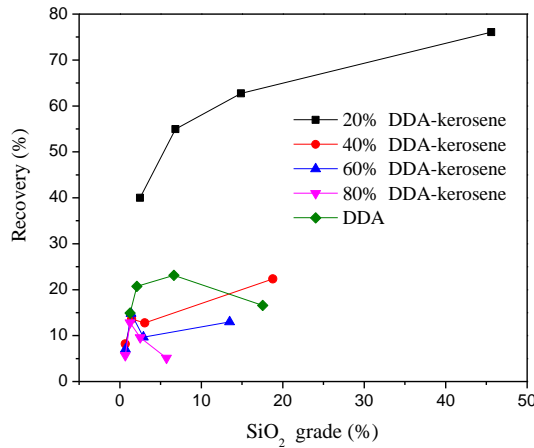


Fig. 12. SiO₂ grade-recovery plot. Grade in the X-axis from low to high was: -0.038, 0.038-0.045, 0.045-0.074, +0.074 mm size fraction

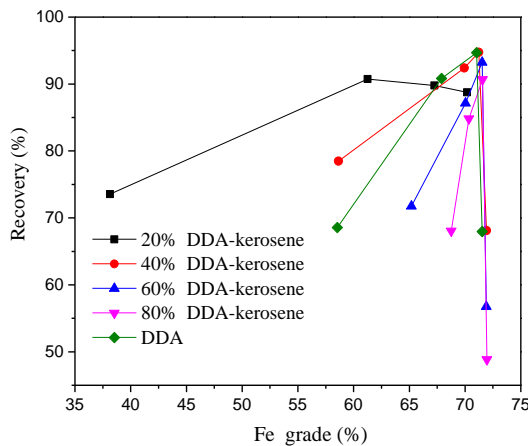


Fig. 13. Fe grade-recovery plot. Grade in the X-axis from low to high was: +0.074, 0.045-0.074, 0.038-0.045, -0.038mm size fraction

The size analysis of the raw ore sample shows that magnetite mainly distributed in -0.038 mm size fraction, and quartz mainly distributed in 0.045~0.074 and -0.038 mm size fractions. Meanwhile the distribution degree of quartz and magnetite was 10.32 and 1.31%, respectively in +0.074 mm size fraction with 2.19% yield. It is clear that the coarse particles that were difficult to float required higher collector dosage. However, higher DDA consumption resulted in more fine fraction (-0.038 mm) magnetite loss. It suggests that the most reasonable technological processes would be: grinding, coarse and fine classification, and regrinding of coarser size fraction (+0.074 mm) and flotation of fine (mainly -0.074 mm) particles with DDA-kerosene.

Conclusions

The study was carried out to investigate the synergistic effect of DDA-kerosene on flotation of magnetite ore and to find the main trends of the impact of DDA-kerosene composition on flotation performance of magnetite ore. The main conclusions were as follows.

The flotation data of pure minerals showed that DDA-kerosene performed better than DDA-HCl, as it had similar collecting capability towards quartz, while relatively higher selectivity towards magnetite. DDA proportion of 50% can be substituted by relatively cheaper kerosene without affecting quartz recovery compared with pure DDA.

During flotation of magnetite ore, fine particles selectively floated with DDA-kerosene. Coarse particles with high SiO₂ content, that were difficult to float, required higher DDA input. Higher concentration of DDA caused losses of magnetite in the fine fraction (-0.038 mm). The most reasonable technological process was classification of coarse and fine particles, regrinding of the coarse fraction, and flotation of fine (mainly -0.074 mm) fraction with a small amount of DDA.

In flotation of magnetite ore, the optimum proportion of DDA is 40% in the mixture of collectors. The concentrate grade increased from 68 to 71% compared with the pure DDA. Magnetite concentrates with 1.83% SiO₂ and 71.04% Fe were produced using 120 g/Mg of DDA-kerosene ($W_{\text{DDA}}=40\%$).

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